65-2959

DETERMINATION OF THE ALUMINA CONTENT OF ALUMINUM ALLOYS

TECHNICAL REPORT

By

Richard B. Miclot

November 1965

U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL

RESEARCH & ENGINEERING DIVISION

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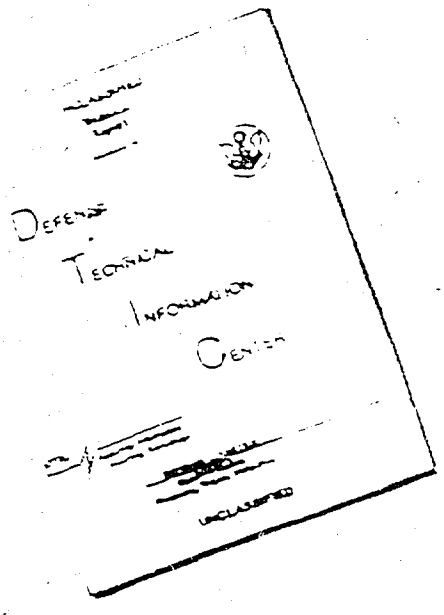

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DETERMINATION OF THE ALUMINA CONTENT OF ALUMINUM ALLOYS

By

Richard B. Miclot
Laboratory Branch

November 1965

DA # 1C014501B32A AMS Code 5011.11.855

ABSTRACT

A method has been investigated for the quantitative determination of aluminum oxide in aluminum alloys. Iodine and chlorine were used as isolating agents for aluminum oxide in an aluminum matrix. A spectrophotometric method of aluminum analysis is presented employing these halogen reagents. Limited test results on aluminum foil and wire indicate that a high iodine concentration in methanol, when chlorine is present, yields consistent results of Al_2O_3 content.

TABLE OF CONTENTS

PROBLEM

The objective of this investigation is twofold:

1. To develop an analytical procedure to determine alumina content in aluminum and aluminum alloys.

2. To determine the effects of specific isolating reagents for alumina in aluminum and aluminum alloys.

BACKGROUND

Heterogeneous impurities in aluminum casting alloys, such as oxide films that accumulate in foundry melts, may frequently be the underlying cause of casting defects, sometimes occurring in epidemic proportions. Considerable evidence exists to show that oxide films entrained with intermetallic compounds create planes of weakness in castings and lower fluidity and the tensile and elongation levels. Large size particles of oxide are detrimental to cast metal, but little is known of the effect of intermediate size and finely divided, colloidal alumina in aluminum castings. A knowledge of concentration, location, and distribution of these fine particles may well improve understanding of the associated mechanical and physical properties in castings.

A reliable method is needed to show whether harmful effects on mechanical and physical properties may be due solely to oxide contamination. Several qualitative criteria of oxide presence have been employed and good mechanical properties have been considered an indicator of cleanliness. However, a dearth of quantitative information exists on alumina in cast aluminum alloys because precise measurements have been intricate and not very dependable. Efforts were made to develop a practical way to determine aluminum oxide in aluminum casting alloys.

APPROACH

The plan adopted for chemical analysis procedure involves dissolution of the metallic constituents, separation, and spectrophotometric determination of the unreacted alumina. Examinations were conducted on separate parts of the procedure. All alumina determinations were obtained via the aluminon method in acid medium, as outlined by Sandell.[1] All special reagent preparations and test measurements, except spectrographic analyses, were made by a single investigator.

The general procedure for sample dissolution is a modification of the approach taken by an early investigator.[2] Various dissolution reagents were prepared and tested on wrought and cast aluminum alloys. The combination of a non-oxide forming dissolvent in a water-free medium was selected so as to avoid possible Al_2O_3 formation and hydration of products. Halogens were examined in several non-aqueous solvents. Methanol was chosen as a satisfactory halogen solvent and was used throughout this work.

Samples of aluminum foil were treated with special reagents. The complete method for reagent preparation and alumina analysis and the spectrographic analysis of the foil are presented in Appendix A.

RESULTS AND DISCUSSION

The analysis for alumina in the foil is given in Table I.

Exothermic reactions occur when aluminum and aluminum alloys are treated with reagents listed in Table I. The chlorinated reagents cause spontaneous vapor bursts after a period of apparent inactivity. High, inconsistent test results may be attributed to these vapor bursts. Rapid reaction can cause spatter and isolation of fine, undissolved metal particles, which are later oxidized. Precautions had been taken in an effort to minimize this condition:

1. Sample and dissolution reagent were kept near room temperature by means of a water jacket.

2. Small quantities of reagent were added intermittently to the sample.

3. Small sample surface area was exposed to the reagent.

4. The reaction beaker was placed in a flushing nitrogen atmosphere.

Iodine readily precipitates from solutions of 59 wt. % iodine chlorinated in methanol. Solid iodine is observed in reagent storage or after sample dissolution.

Liquid iodine monochloride, ICl, was considered as an isolating agent for alumina. Compound preparation is accomplished by direct chlorine-to-iodine contact. Small specimens of aluminum wire or cast aluminum alloys ignite

TABLE I

ALUMINA CONTENT OF ALUMINUM FOIL DISSOLVED BY HALOGENS IN METHANOL

Iodine Chlorinated in Methanol Wt. % Iodine	Equivalent Wt. % Iodine Monochloride	% Al_2O_3 Found	Avg. % Al_2O_3 Found	Standard Deviation	% Coefficient Variation
11	13	0.46, 0.18, 0.34, 0.24	0.305	0.122	40
20	24	0.21, 0.10, 0.21, 0.18	0.175	0.051	29
35	41	0.13, 0.16, 0.18, 0.15	0.155	0.020	13
54	60	0.08, 0.09, 0.11, 0.10	0.095	0.012	13
59	65	0.08, 0.08, - -	0.080	-	-

when contacted with ICl at the liquid surface. Early investigators of this interhalogen have reported that aluminum and other elemental materials react explosively with ICl.[3] This hazardous condition presently discourages further investigation of the compound at 100% concentration.

Aluminum wire, analytical grade, was submitted to attack by the halogen-in-methanol reagents. Test results are shown in Table II. Spectrographic analysis of the wire is given in Appendix A.

The chlorine reagent is prepared in the presence of the sample. Chlorine gas flow in the methanol is regulated by equipment described in the literature.[4] During dissolution reaction fine, disintegrated particles of the attacked sample can be seen rising to the surface of the iodine-free, transparent liquid. After apparent dissolution of sample a gray residue is retained on the filter. High alumina test results are obtained when such conditions occur. The cause of higher apparent alumina content is attributed to incomplete sample dissolution.

Samples of aluminum foil and added alumina were treated with prepared halogen reagents. The alumina was prepared in fine particle size after thermally decomposing aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$. This compound is selected as a reliable source of chemically inactive alumina. Mild heating in air readily releases H_2O vapor from the hydrate. NO_2 gas (brown fumes), and O_2 escape with higher heat application leaving the alumina residue:

$$4\left[Al(NO_3)_3 \cdot 9H_2O\right] \rightarrow 36H_2O\uparrow + 12NO_2\uparrow + 3O_2\uparrow + 2Al_2O_3\downarrow$$

Gravimetric tests show that conversion to the oxide by heating is greater than 99.0% complete. Weight-loss tests also show that less than 0.30% of the calcined nitrate is lost when exposed to the alcoholic halogen reagents.

The reagent effects on foil with added oxide are shown in Table III in terms of oxide recovered. A mineral acid in aqueous solution is included in the comparison of the dissolving reagents.

Data in Tables II and III indicate higher apparent alumina contents when metal samples are exposed to monohalogen reagents. A similar trend in total alumina content is observed when anodized aluminum wire is treated with these reagents.

TABLE II

ALUMINA CONTENT OF ALUMINUM WIRE DISSOLVED BY HALOGENS IN METHANOL

Dissolution Reagent in Methanol	% Al_2O_3 Found	Avg. % Al_2O_3	Std. Dev.	% Coefficient of Variation
11 Wt. % Iodine	0.46, 0.22, 0.40, 0.28	0.340	.109	32
54 Wt. % Iodine, Chlorinated	0.08, 0.14, 0.11, 0.14	0.117	.028	24
Chlorine	0.72, 0.80, 0.42, 0.94	0.720	.219	30

TABLE III

THE EFFECT OF DISSOLUTION REAGENTS ON ALUMINA RECOVERY FROM ALUMINUM FOIL WITH ADDED ALUMINA

Dissolution Reagent	% Al_2O_3, After Addition to Foil	Apparent % Al_2O_3 Found	% Yield
1:1 Hydrochloric Acid	3.94	1.68	42
	1.95	1.21	62
	1.63	0.82	50
11 Wt. % Iodine in Methanol	2.81	2.66	95
	3.98	3.90	98
	0.69	1.83	265
54 Wt. % Iodine Chlorinated in Methanol	1.19	1.15	96
	0.98	1.10	112
	1.19	0.99	83
Chlorine in Methanol	0.64	1.63	255
	1.28	2.67	208
	1.01	1.89	187

Alcoholic halogen attack on casting alloys was examined. Iodine in methanol, 11 wt. % iodine, was used on cast samples of 356, C355, Almag 35, 220, 195, 40E, and Precedent 71A alloys. Each of the alloys is reactive with this halogen, but complete dissolution of metallic constituents is questionable.

A residue from the 356 alloy was isolated after dissolution treatment. X-ray diffraction measurements identified the material principally as elemental silicon. When an ashed residue from this Al-Si alloy is treated with conc. H_2SO_4 and a few ml. of HF, a gray lustrous film envelops the remaining solid. The film is presumed to be metallic aluminum. During carbonate fusion flame bursts are observed with the disappearance of the film. Occlusion of aluminum by insoluble silicon particles and subsequent oxidation of aluminum are suspected as the cause of the high alumina values. Extracted residues from the 220 alloy, (10% Mg), and Precedent 71A alloy, (7% Zn), were examined by the X-ray diffraction method. Positive identification of residues was not confirmed. These low silicon, probably spinel-type residues are likewise suspected as contributors to aluminum occlusion leading to high alumina results.

Presently, 356 and the other casting alloys have not been exposed in quantitative tests to attack, either by chlorinated iodine in methanol, or by chlorine in the alcohol. Although silicon has been reported as unattacked by iodine monochloride,[3] perhaps the occluded aluminum, or alumino-intermetallic compounds, can be dissolved by this interhalogen.

After metallic dissolution is effected, the unreacted alumina is rendered soluble by acid or alkali fusion. A study was made on the fusion operation to convert alumina to a soluble meta-aluminate. A fusion mixture was prepared similar to the reagent used by other investigators.[5] Portions of calcined aluminum nitrate were fused with the prepared alkali mixture. Aliquots from the acidified extracts were analyzed. The test results are reported in Table IV in terms of micrograms of Al_2O_3.

Measurements indicated in Tables III and IV are exposed to probable error due to small sample weight.

Ferric iron causes color interference in the aluminon method. The alkali fusion reagent selected for this investigation has been used by others to eliminate iron as a water-insoluble product.[5] Some searchers, employing acid fusion, compensate by making requisite iron additions to match an iron-in-alumina calibration.[6]

TABLE IV

% CONVERSION OF Al_2O_3 BY Na_2CO_3-$Na_2B_4O_7$ FUSION

Na AlO_2 Aliquot Equivalent μg Al_2O_3	Na AlO_2 Found Equivalent μg Al_2O_3	% Conversion
60	59	98
124	107	86
62	59	94
49	48	97
96	82	85
192	169	88
190	169	89
95	85	89
120	103	86
120	101	84
180	153	85
282	241	85
113	90	79
119	102	86
182	162	89
88	77	87
177	160	90
113	100	88
	Mean	88
		Std. Dev. ± 4.6

1μg = 1 Microgram = 1 X 10^{-6} Gram

Thorough investigation remains to be completed for iron elimination or control. However, the effect of ferric iron in the final volumetric solution has been observed in the current study. Increase in iron-to-alumina weight ratio produces higher apparent alumina concentration, (lower transmittance). This iron effect is more pronounced at higher alumina content.

Tests were conducted to determine the relation between the error in transmittance measurement and the resulting error in concentration. Dilutions were made from a standard calibration solution. 10 ml. aliquots from the dilutions were tested to determine the variation in transmittance with invariant alumina concentrations. A summary of the test results appears in Table V. Test data and computations are in Appendix B.

Gain in precision results when the sample size increases. Thus, a sample average for 3 tests produces $1/\sqrt{3} = 0.577$, or 58% of the confidence limits spread for individual tests. The narrower limits exhibit less variation in concentration values.

A semilogarithmic plot, % transmittance vs $\mu g\ Al_2O_3/ml$, is shown in Figure 1, Appendix B. The calculated best fit line is plotted with individual transmittance measurements. Concentration values derived from 95% confidence limits indicate a uniform variation of $\pm$ 0.3 $\mu g\ Al_2O_3/ml$ for all individual transmittance measurements. Duplicate tests reduce the variation to approximately $\pm$ 0.2 $\mu g\ Al_2O_3/ml$. At the concentration level of 4.0 $\mu g\ Al_2O_3/ml$ tests in duplicate show $\pm$ 5% error involved in the comparison of color intensity. At 0.5 $\mu g\ Al_2O_3/ml$ a $\pm$ 42% error results.

An overall check of the analytical procedure has not been accomplished in the absence of reliable oxide-in-metal standards. Trial melts were prepared in an effort to develop suitable standards. However, injected solid oxide in molten aluminum is not retained in uniform distribution, even when the treated metal is rapidly solidified. Attempts at air-lancing aluminum melts were unable to create uniform oxide dispersion in the solid state.

Anodized aluminum wire is considered as a possible source to provide a constant weight ratio of oxide to metal. Early trials to produce a uniform alumina deposit over a sufficient length of wire were unsuccessful.

Nonuniformity in these investigations is judged by the gross differences of total alumina content found by the aluminon method.

TABLE V

% TRANSMITTANCE VARIATION WITH FIXED ALUMINA CONCENTRATION

No. Aliquots Tested	$\mu g\ Al_2O_3$ Per Ml	Arithmetic Mean % Transmittance	Mean From Best Fit Line % Transmittance	95% Confidence Limits Individual Tests % Transmittance	
				Upper	Lower
11	0.4	82.2	83.4	93.5	74.3
19	1.0	66.1	66.2	74.3	59.0
15	2.0	46.5	45.1	50.6	40.2
17	3.0	31.1	30.7	34.4	27.0
13	4.0	20.4	20.9	23.4	18.6

CONCLUSIONS

1. A method has been investigated for determining aluminum oxide content in aluminum. The method appears adequate for the practical analysis of aluminum castings and for the study of the effect of oxide content on their mechanical properties. Accuracy and reliability of the method have not yet been completely established because of a lack of suitable standards.

2. Limited test results indicate that greater consistency is obtained by the method when both iodine and chlorine are used in the dissolution reagent. Less variation in results occurs if high concentrations of the dihalogen reagent are employed.

RECOMMENDATIONS

The overall method requires further analytical evaluation and determination of its reliability and precision. The following measures are suggested:

1. Develop an alumina-metal standard, such as anodized aluminum wire, that provides a constant weight ratio of oxide to metal.

2. Determine reliability and precision of the method from test results obtained at selected levels of oxide content in standards.

3. Appraise the method with other analytical procedures by comparing test results obtained on a common standard.

A reagent is required to completely dissolve occluded aluminum in aluminum alloys. Alcoholic iodine-chlorine reagent of high halogen concentration is recommended as a potential dissolvent.

A practical application is suggested based upon method acceptance:

Determine what alumina concentration level is critical in its effect on the mechanical properties of castings.

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APPENDIX A

ANALYSIS BY ALUMINON METHOD

DISSOLUTION REAGENT PREPARATION

In an exhaust hood pass dry chlorine into a cold water-jacketed beaker containing 93 grams of iodine per 100 ml methanol. Metered chlorine flow should be regulated to permit fine bubble dispersion and minimum escape of free chlorine. During reaction the iodine solids dissolve and the liquid develops a dark red, liquid bromine color. Complete reaction is assumed when a stoichiometric amount of chlorine has been transferred to the iodine solution to form iodine monochloride. This chlorinated solution is equivalent to 60 wt. % iodine monochloride, or 54 wt. % iodine. Store reagent at room temperature in an exhaust hood in a plastic-capped glass bottle.

DISSOLUTION AND SEPARATION

Weigh a 0.5 to 1.0 gram sample of aluminum or aluminum alloy, (small surface area preferred), into a 300 ml high form beaker. Transfer beaker to an exhaust hood and add 60 to 70 ml chlorinated iodine-methanol reagent. Place watchglass over beaker and set beaker in cooling tray. After reaction eases, spray down watch-glass and beaker walls with methanol. Suction-filter beaker contents through Schleicher-Schuell #589 Red Ribbon filter paper, or its equivalent. Wash filter with methanol and dry paper at 110°C for 30 minutes. Ash paper in platinum crucible. Thoroughly mix the residue with 3 grams of $K_2S_2O_7$ or 1 gram Na_2CO_3-$Na_2B_4O_7$ mixture. Carefully heat to fuse, avoiding spatter, until clear liquid develops. Air cool crucible and leach residue in a small amount of hot water, less than 80 ml. Rinse crucible and filter the liquid into an appropriate volumetric flask containing 5 ml 1:4 HCl, if Na_2CO_3 fusion was performed. Adjust pH of solution to slightly less than 7.0 and dilute to mark.

PHOTOMETRIC PROCEDURE

Transfer a 1 to 10 ml aliquot sample to a 50 ml volumetric flask. Pipette 2 ml of 1:9 HCl and 2 ml of 0.2% aluminon solution into the flask. Dilute to about 30 ml. Pipette in 10 ml of 10% ammonium acetate and dilute to mark. Transfer some solution and reagent blank to matched cells. Set reference to 100% transmittance at 520 mμ. Read sample transmittance 18-20 minutes after last reagent addition. Obtain aluminum oxide content from a

plot of % transmittance vs. micrograms aluminum oxide.

APPARATUS

Bausch and Lomb colorimeter - Spectronic 20

REAGENTS AND SOLUTIONS

Iodine, analytical reagent Non volatile matter - 0.020%
Chlorine, liquefied, high purity, 99.5% min.
Methanol, technical, 99.85%
Fusion mixture - 3 parts Na_2CO_3, 1 part $Na_2B_4O_7$
Na_2CO_3 0.001% Fe
$Na_2B_4O_7$ 0.0001%
Potassium pyrosulfate, $K_2S_2O_7$, fused powder 0.001% Fe
Aluminon - 0.2% solution of ammonium aurintricarboxylate in water.
Ammonium acetate - 10% solution in water
Aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, 0.002% Fe
Standard Solution for Curve Calibration:
Aluminum potassium sulfate, $KAl(SO_4)_2 \cdot 12H_2O$, 0.0001% Fe dissolved in 0.1 normal HCl (equivalent to 100 μg Al_2O_3 per ml).

SPECTROGRAPHIC ANALYSES OF METAL SAMPLES

	Cu	Fe	Si	Mn	Ti	Mg	Zn	Cr
Aluminum foil, %	.07	.35	.10	<.01	<.01	.02	.02	<.01
Aluminum wire, %	.02	.10	.10	.001	.03	-	-	-

APPENDIX B

TRANSMITTANCE MEASUREMENTS FOR VARIOUS DILUTIONS FROM A STANDARD SOLUTION

T - % transmittance reading, C = $\mu g\ Al_2O_3/ml$

C = 0.4		C = 1.0		C = 2.0		C = 3.0		C = 4.0	
T	$\log_{10} T$	T	$\log_{10} T$	T	$\log_{10} T$	T	$\log_{10} T$	T	$\log_{10} T$
83	1.919	67	1.826	45.5	1.658	30.5	1.484	23	1.361
81	1.908	64	1.806	44	1.643	29.5	1.469	18	1.255
83	1.919	66	1.819	48	1.681	33	1.518	20.5	1.311
81	1.908	66	1.819	48	1.681	33	1.518	22	1.342
82	1.913	68	1.832	49	1.690	32	1.505	21	1.322
81.5	1.911	68	1.832	475	1.676	32	1.505	21	1.322
82	1.913	67	1.826	47	1.672	32	1.505	23	1.361
83	1.919	67	1.826	47.5	1.676	31.5	1.498	23	1.361
83	1.919	68	1.832	47.5	1.676	32	1.505	19	1.278
82.5	1.916	66.5	1.822	45.5	1.658	34	1.531	19	1.278
82.5	1.916	67	1.826	46	1.662	34	1.531	19	1.278
		67.5	1.829	45.5	1.653	29.5	1.469	19	1.278
		67	1.826	46	1.662	30	1.477	19	1.278
		65.5	1.816	45	1.653	29.5	1.469		
		65	1.812	45.5	1.658	29.5	1.469		
		65	1.812			28	1.447		
		65	1.812			29.5	1.469		
		64.5	1.809						
		64	1.806						

APPENDIX B (Continued)

The best fit line for the transmittance data is derived by a statistical method, assuming that Beer's Law for light transmittance holds.[7] The line equation for the data is:

$$\text{Log } T = 1.988 - 0.167c$$

where T = % transmittance,

and c - μg Al_2O_3/ml

Confidence limits for individual transmittance measurements are obtained by Hader's method of analysis of variance.[8] A summary is tabulated.

95% CONFIDENCE LIMITS FOR INDIVIDUAL TRANSMITTANCE VALUES

Selected c	T Point Std. Dev.	Best Fit Line log T	Best Fit Line T	95% C.L. Upper log T	95% C.L. Upper T	95% C.L. Lower log T	95% C.L. Lower T
0.2	0.025	1.955	90.1	2.005	101.2	1.905	80.4
0.4	0.025	1.921	83.4	1.971	93.5	1.871	74.3
1.0	0.025	1.821	66.2	1.871	74.3	1.771	59.0
2.0	0.025	1.654	45.1	1.704	50.6	1.604	40.2
3.0	0.025	1.487	30.7	1.537	34.4	1.437	27.0
4.0	0.025	1.320	20.9	1.370	23.4	1.270	18.6
5.0	0.026	1.153	14.2	1.205	16.0	1.101	12.6
7.0	0.027	0.819	6.6	0.873	7.5	0.765	5.8

Concentration variation with transmittance confidence limits is determined from the slope of the best fit line

$$C_u - C_l = \frac{\log T_u - \log T_l}{.167},$$

where C_u = μg Al_2O_3/ml for lower transmittance limit, T_l,

and C_l - μg Al_2O_3/ml for upper transmittance limit, T_u.

CONCENTRATION VARIATION WITHIN 95% CONFIDENCE LIMITS FOR INDIVIDUAL TRANSMITTANCE VALUES

c	log T_u	log T_l	log T_u-log T_l	C_u-C_l
0.4	1.971	1.871	0.100	0.6
1.0	1.871	1.771	0.100	0.6
2.0	1.704	1.604	0.100	0.6
3.0	1.537	1.437	0.100	0.6
4.0	1.370	1.270	0.100	0.6

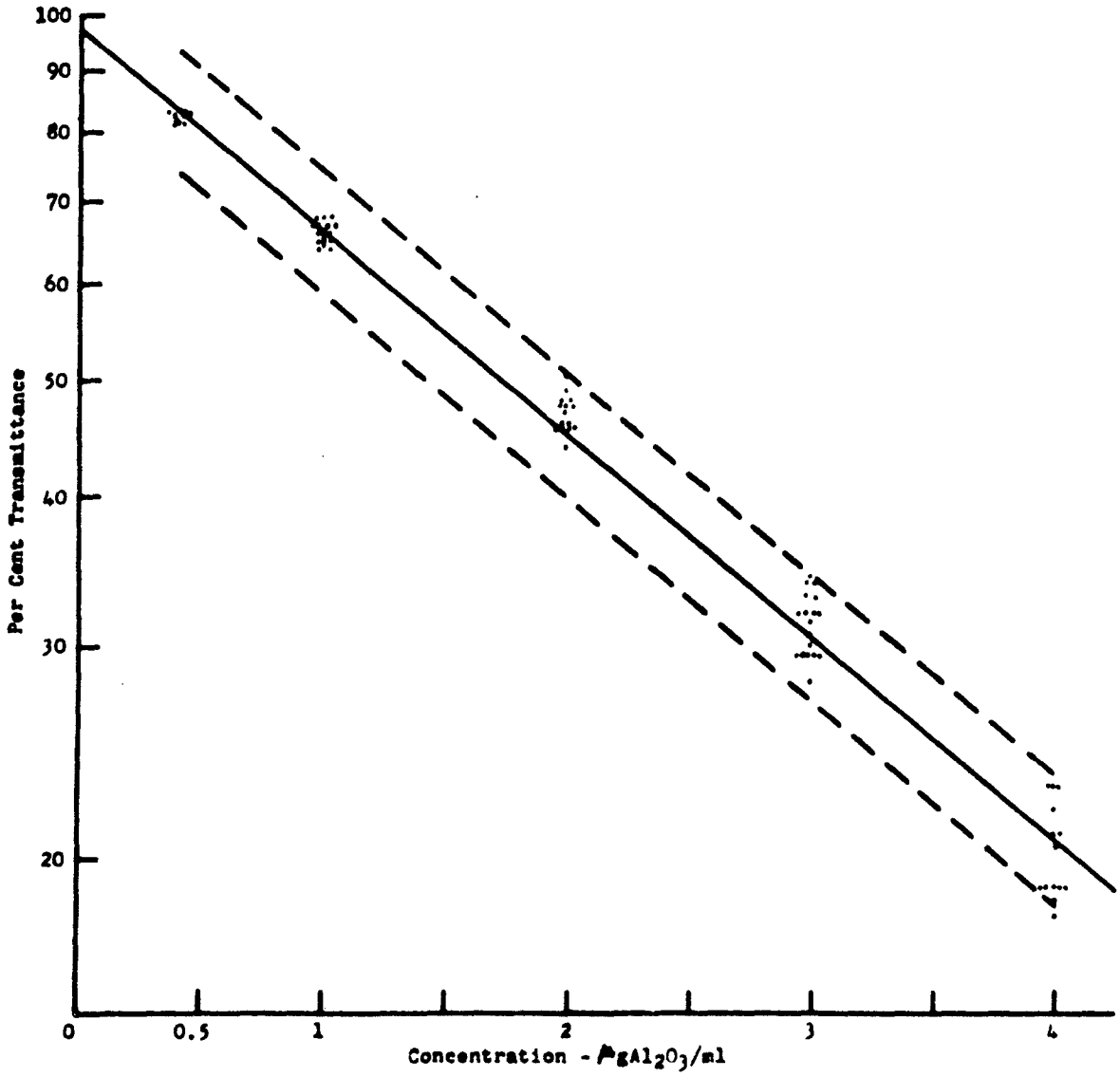

FIG. 1

TRANSMITTANCE MEASUREMENTS, BEST FIT LINE, AND 95% CONFIDENCE LIMITS FOR VARIOUS ALUMINA CONCENTRATIONS

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